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Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

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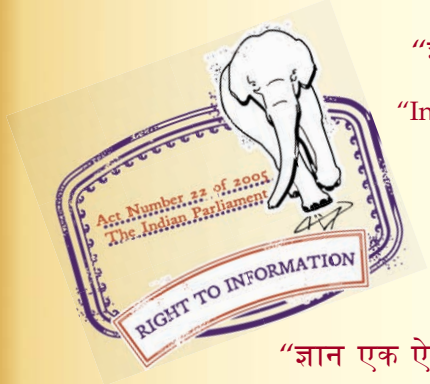
IS 1448-84 (1975): Methods of test for petroleum and its products, Part 84: Determination of trace elements in Petroleum products -- Arsenic [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]



"ज्ञान से एक नये भारत का निर्माण"
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"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

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Indian Standard

(Reaffirmed 2006)

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 84]

DETERMINATION OF TRACE ELEMENTS IN PETROLEUM PRODUCTS — ARSENIC

(Second Reprint APRIL 1988)

UDC 665·6/·7 : 543·064 [546·19]

Adapted from International Series of Monographs on Ana lytical Chemistry, Volume 14 Analysis of Petroleum for Trace Elements by O. T. Milner, published by PERGAMON PRESS, LONDON, 1963

1. SCOPE

1.1 This method is intended for the determination of arsenic in petroleum naphthas and other volatile distillates. The procedure is a rapid one and requires about 2 h in elapsed time. About 100 ml of sample is used and it is accurate over the range of 1 to 100 ppb.

2. OUTLINE OF THE METHOD

2.1 Arsenic in the sample is extracted with sulphuric acid and hydrogen peroxide. The extract is digested with hydrogen peroxide to destroy the organic matter and the resulting solution is then transferred into an Erlenmeyer flask for arsine generation (*see* Fig. 1). Potassium iodide, stannous chloride, and zinc are added and the arsine so evolved is then absorbed in silver diethyldithiocarbamate solution in pyridine. Arsenic forms a coloured complex with silver diethyldithiocarbamate and it is determined colorimetrically.

3. APPARATUS

3.1 Arsine Evolution Apparatus — consisting of evolution flask, hydrogen sulphide scrubber and arsine absorber as shown in Fig. 1.

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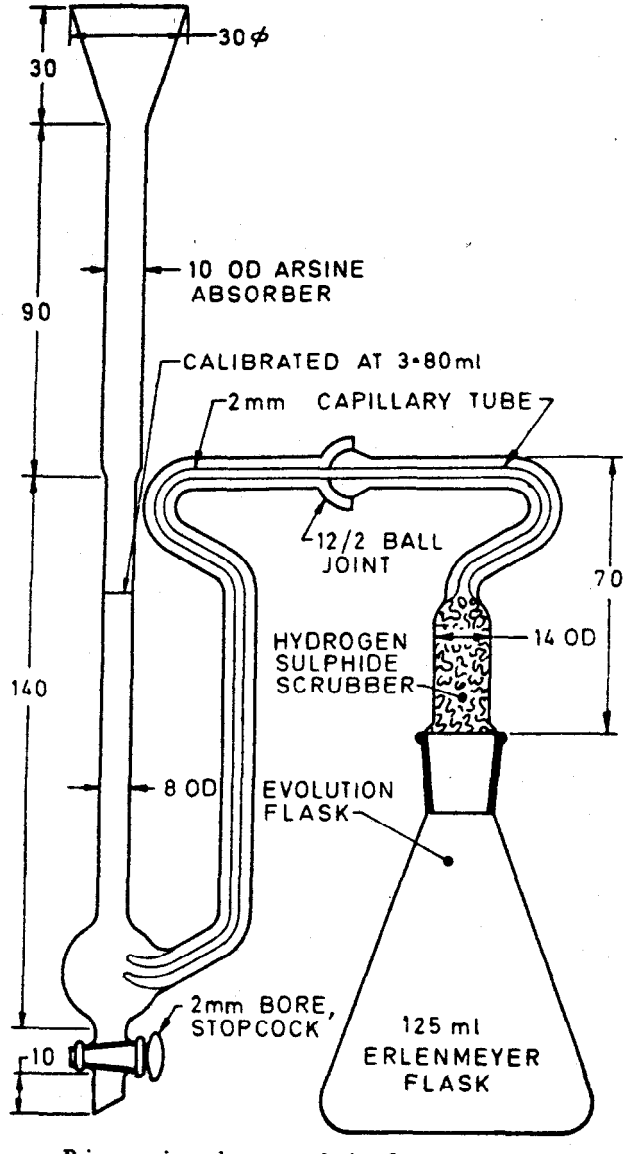

Bring receiver close to evolution flask for stability.

All dimensions in millimetres.

FIG. 1 ARSINE EVOLUTION APPARATUS

3.2 Spectrophotometer* — capable of measuring absorbance in the region of 540 nm and provided with matched cells of 70 mm. If decreased sensitivity is required, ordinary 20 mm cells of 5 ml total capacity may be used; in this case the total volume of the absorbing solution is adjusted to 5·00 ml instead of 3·80 ml.

3.2.1 Alternatively, a suitable photoelectric colorimeter may be used in place of spectrophotometer.

3.3 Kjeldahl Flask — 500-ml capacity.

4. REAGENTS

4.0 Quality of Reagents — Unless otherwise specified, pure chemicals and de-ionized water shall be used in the test. Water shall be de-ionized by passing through a mixed bed ion-exchanger.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

4.1 Sulphuric Acid — of 96 percent purity (relative density 1·84) having arsenic content 0·000 01 percent, *Max*.

4.2 Standard Arsenic Solution

4.2.1 *Standard Arsenic Solution* (0·100 *mg Arsenic per ml*) — Dissolve 0·132 g of arsenic trioxide in 2·5 ml of 20 percent sodium hydroxide, acidify with 2·5 ml of dilute sulphuric acid (1 : 5) and dilute to 1 000 ml with water.

CAUTION — Arsenic compounds are highly toxic. Do not apply mouth suction when pipetting solutions. Prevent contact with skin and eyes. Avoid inhalation of dusts and fumes.

4.2.2 *Dilute Standard Arsenic Solution* (0·1 μg *Arsenic per ml*) — Dilute 1 ml of standard arsenic solution (0·100 mg arsenic per ml) to 1 000 ml with water immediately before use.

4.3 Zinc Metal, Reagent Grade, Granular, 850-micron Size — The arsenic content of this reagent varies widely. A lot containing preferably less than 0·3 μg per 5 g, as determined by blank runs with slightly varying amounts of zinc, should be reserved for this determination.

4.4 Silver Diethyldithiocarbamate (AgDDC) — Prepare two solutions, one containing 1·7 g of silver nitrate in 100 ml of water and the other containing 2·3 g of sodium diethyldithiocarbamate in 100 ml of water. Cool below 20°C and mix slowly with stirring. Filter off the lemon-yellow precipitate of silver diethyldithiocarbamate (AgDDC) and wash

*Beckman Model B or any other suitable instrument may be used.

thoroughly with distilled water. Dry in a vacuum desiccator. The dry salt is stable at room temperature for at least six months. The reagent is also commercially available.

4.5 Silver Diethyldithiocarbamate Solution — Dissolve 1·0 g of the salt in 200 ml of pyridine. Allow the solution to age for at least a week before use in order to ensure reproducible calibration. Alternatively, a calibration curve may be prepared at the time that unknown samples are analyzed. Once aged, the reagent will remain stable for several months.

4.6 Lead Acetate-Coated Glass Wool — Soak glass wool in 10 percent lead acetate solution, drain well, and dry. Store in a capped jar.

4.7 Stannous Chloride Solution — Dissolve 2 g of reagent in 10 ml of concentrated hydrochloric acid. Prepare fresh at least once a week.

4.8 Potassium Iodide — 15 percent solution.

4.9 Hydrogen Peroxide — 30 percent.

4.10 Pyridine

5. PROCEDURE

5.1 Add 15 ml of concentrated sulphuric acid and 20 ml of hydrogen peroxide to a 500-ml Kjeldahl flask containing one or two pumice stones, and cool to 0 to 5°C. Add up to 100 ml of sample containing up to 1 μg of arsenic to the flask. Some samples, especially those which are high in olefins, react vigorously with the acid-peroxide mixture. Therefore, shake the mixture gently at first. If a vigorous reaction is apparent or if the bottom of the flask heats up quickly, plunge the flask into ice water until the reaction subsides. If the mixture darkens at any time, add more hydrogen peroxide. Shake the mixture vigorously for 1 minute, then reflux the sample for half an hour with occasional shaking.

5.2 Cool the mixture, transfer it to a separatory funnel, and drain the acid layer back into the Kjeldahl flask, washing the hydrocarbon layer once with 10 ml of water. With samples that do not char easily and with samples of 10 ml or less, the separation may be eliminated and the organic layer boiled away.

5.3 Heat the solution to sulphuric acid fumes, adding hydrogen peroxide when necessary to prevent excessive charring. Cool the solution, wash it into an evolution flask, and adjust the total volume to 65 $\pm$ 5 ml. Cool the solution to 20°C and add 1 ml of potassium iodide solution and, with swirling, 5 or 6 drops of stannous chloride solution. Add 5·0 $\pm$ 0·1 g of zinc metal and immediately attach the hydrogen sulphide scrubber and the absorber containing 3·80 ml of silver diethyldithiocarbamate (AgDDC) solution.

5.4 Allow the evolution to proceed for 30 minutes, then detach the absorber and dilute the solution again to exactly 3·80 ml with pyridine to replace what is lost during evolution. Pass a gentle air stream through the absorber for a few seconds to mix the solution. Immediately measure the absorbance at 540 nm in the 70 mm cell; use silver diethyldithiocarbamate (AgDDC) solution as a reference. Convert the absorbance to μg (microgram) of arsenic by referring to a calibration curve that has been prepared by evolving arsine from known amounts of arsenic in the range of 0·1 to 1 μg starting with 15 ml of sulphuric acid diluted to 65 ml.

6. CALCULATION AND REPORTING

6.1 Calculate the arsenic content as follows:

$$\text{Arsenic, parts per billion (ppb)} = \frac{M \times 10^3}{V \times D}$$

where

M = mass in μg (micrograms) of arsenic obtained from calibration curve after proper blank correction;

V = volume in ml of the sample taken for the analysis; and

D = relative density of the sample.

6.2 Report the result as arsenic content parts per billion.

7. PRECISION

7.1 The precision limits of this method have not yet been established.

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